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CONTROLLING SHAPES AND SIZES OF SYNTHESIS SILVER NANOWIRES BY POLYOL METHOD USING POLYVINYL ALCOHOL AND POLYVINYL PYRROLIDONE

PENGUSUL

(Dr. Junaidi, S.Si., M.Sc. NIDN: 0018068204)

JURUSAN FISIKA
FAKULTAS MATEMATIKA DAN ILMU PENGETAHUAN ALAM
UNIVERSITAS LAMPUNG
2017

HALAMAN PENGESAHAN
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b.	NIDN	:	0018068204
c.	Jabatan Fungsional	:	Asisten Ahli
d.	Program Studi	:	Fisika
e.	Nomor HP	:	0857-5888-3337
f.	Alamat surel (e-mail)	:	Jurusan Fisika FMIPA Universitas Lampung Jl. Sumantri Brojonegoro No. 1 Gedong Meneng Bandar Lampung 35245

II. IDENTITAS JURNAL (Tempat terbit artikel ilmiah yang diusulkan)

a.	Nama Lengkap Jurnal	: Indian Journal of Science and Technology
b.	Nama Singkatan Jurnal	: INDJST
c.	Nomor ISSN	: 0974-5645
d.	Cakupan Bidang Ilmu	: Multidisiplin (Science and Technology)
e.	Alamat Redaksi	: Indian Society of Education and Environment, India
f.	Penerbit	: Informatics Publishing Limited
g.	Vol/Number terbit	: Vol. 10, No. 27
h.	Tgl/bln/Thn terbit	: Juli 2017
i.	Lembaga Pengindeks	: DOAJ, Thomson Router, EBSCO, Scopus
j.	Nilai Impact Factor	: 0,25
k.	Alamat website jurnal	: http://www.indjst.org/index.php/indjst/issue/view/7438

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ISSN (Print) : 0974-6846
Indian Journal of Science and Technology, *Vol 10(27), DOI: 10.17485/ijst/2017/v10i27/93895, July 2017* ISSN (Online) : 0974-5645

Controlling Shapes and Sizes of Synthesis Silver Nanowires by Polyol Method using Polyvinyl Alcohol and Polyvinyl Pyrrolidone

Junaidi[1,2], Kuwat Triyana[2*], Harsojo[2] and Edi Suharyadi[2]

[1]Department of Physics, Universitas Lampung, Bandar Lampung, 35145 Indonesia
[2]Department of Physics, Universitas Gadjah Mada, Yogyakarta, 55281 Indonesia;
junaidi.1982@fmipa.unila.ac.id, triyana@ugm.ac.id

Abstract

Background/Objectives: To synthesis silver nanowires with Polyvinyl Alcohol (PVA) and Polyvinyl Pyrrolidone (PVP) as a capping agent by the polyol method. **Methods/Statistical Analysis:** Synthesis of silver nanowires was done by adding PVA and PVP for controlling of Multi-Twinned Particles seeds (MTPs) before grown to silver nanowires. The silver nanowires were characterized by UV-vis, SEM and XRD techniques. PVA and PVP is to be absorbed on the surface of the Ag seeds through Ag-O bond to form silver nanowires. Reaction time and temperature, as well as the kind of capping agent, affected the morphologies and sizes of the silver nanowires. **Findings:** The UV-vis spectra of silver nanowires show that the absorbance peaks at a wavelength of 350 to 390 nm. SEM images showed the selective absorption of PVA and PVP on the side surfaces of {100} and {111} facets plays an important role in the growth of anisotropic silver nanostructures. The diameter and length of silver nanowires of PVA were (190 ± 10) nm and (80 ± 10) µm. The addition of PVP as a capping agent can decrease the diameter and length of silver nanowires about 100 nm and 10 to 20 µm, resfectively. XRD pattern of silver nanowires represented that the final product was highly crystallized. The crystal structurecan be identified as a face-centered cubic (fcc) with lattice constant according to the spacing distance between the {111} planes was 4.1454 Å for PVA and 4.0756 Å for PVP. **Applications/Improvements:** PVA can be used as a capping agent for the synthesis of silver nanowires with high aspect ratio. The silver nanowires are synthesized with PVA longer than PVP.

Keywords: Capping Agent, Polyvinyl Alcohol, Polyvinyl Pyrrolidone, Silver Nanowires

1. Introduction

Nanoscience and nanotechnology are the development of science that involves the synthesis and development of nanoscale materials below 100 nm[1,2]. Changes in the size of the materials from micro into nanoscale can change the material properties which can affect the performance of the device or product[3].

The main focus of researchers and industry is the synthesis of silver nitrate ($AgNO_3$) into silver nanowires. The silver nanowires are used as the main material for the transparent electrode for manufacturing optoelectronic device applications. The transparent electrodes are applied for optoelectronic devices, such as organic solar cells, touch screens, transparent heaters and organic light emitting diodes[4–8]. The transparent electrodes made of silver nanowires has equivalent conductivity to ITO[9].

Increasing the number of transparent electrodes based on silver nanowires leads researchers and industries for developing of the synthesis silver nanowires. The latest study is focused on the addition of various precursors such as chloride ions for controlling the size and morphology of silver nanowires[10–12]. The researchers also tried a variety of solvents on the synthesis of AgNWs, such as by using ethylene glycol[13–16], aqueous solution[17,18], 1,2-propendiol[10], glycerol[19,20] and propylene glycol[21]. Until now, there has been no research on the synthesis silver nanowires by using different capping agent besides of Polyvinyl Pyrrolidone (PVP). The researchers only tried the effect of molecular weight and concentration of PVP on the synthesis silver nanowires[22–24].

* *Author for correspondence*

In this study, we tried to combine Polyvinyl Alcohol (PVA) and PVP as a capping agent in the synthesis of silver nanowires. PVA and PVP are polymers have similar physical properties, it can use as a capping agent on the synthesis silver nanowires. As PVP, PVA is a polymer for use as a capping agent on the formation of silver nanowires. For this purpose, PVA may be the best alternative materials as a capping agent for synthesizing silver nanowires. PVA is cheaper and higher mechanical properties compared to PVP[25–27]. PVA is a polymer composed of monomer N-vinyl alcohol (CH_2-CH-OH) easily soluble in water and alcohol. PVA as a capping agent also has many advantages, PVA is non-toxicity, a biocompatible, have high mechanical strength, low membrane permeability, high dielectric constant, and the ability to form a good film. PVA has electrical conductivity value of 9.73×10^{-9} S.cm^{-1} at a temperature of 303 K[28,29]. The presence of PVA can control the morphology and size of the silver nanowires. High mechanical properties of PVA, silver nanowires were synthesized using PVA will be Ag-O bond stronger. The silver nanowires of PVA are not easily damaged and broken when sonicated before deposited on the substrate. For that, we do the synthesis silver nanowires by using PVA and PVP as a capping agent and how to mix both of them for controlling morphology, shapes and sizes of silver nanowires.

2. Materials and Method

2.1 Materials

The materials used for synthesizing of silver nanowires through polyol method included silver nitrate ($AgNO_3$, 99%, Merck), Polyvinyl Alcohol (PVA, Mw. 31000-50000 g/mol, Sigma-Aldrich), Polyvinyl Pyrrolidone (PVP Mw. 55000 g/mol, Sigma-Aldrich), Ethylene Glycol (EG, 99%, Merck), Sodium Chloride (NaCl, 98%, Merck) and Ethanol (EtOH, 98%, Merck).

2.2 Method

2.2.1 Synthesis Silver Nanowires by using PVA as Capping Agent (sample 1)

Firstly, PVA of 0.88 g was dissolved in 20 mL of ethylene glycol into an Erlenmeyer flask immersed in controllable magnetic stirrer oil bath for 15 minutes. Then, 0.5 mL of 10 mM NaCl/EG solutionwas added into the solution. After that, five mL of a 0.5 M $AgNO_3$/EG was added dropwise using a syringe for about 10 minutes, followed by stirring the mixture of these solutions at 700 rpm for 1 hour. The color changed to yellow and became brownish gray after $AgNO_3$/EG added. The solution containing produced of silver nanowires then cooled naturally to room temperature. It followed by being separate with ethanol through several times of centrifugation at a speed of 6000 rpm.

2.2.2 Synthesis Silver Nanowires by using PVP as Capping Agent (sample 2)

Firstly, mix 0.80 gr of PVP and 40 µl of a 0.1 M NaCl in 16 ml of ethylene glycol using an Erlenmeyer flask for one hour. Furthermore, 5 ml of a 0.3 M $AgNO_3$/EG was injected into PVP solution within 3-5 minutes. The stirring process the solution was kept for 90 minutes at a constant temperature of 130°C. The solution containing produced silver nanowires of this process was then cooled naturally to room temperature. It followed by being washed with ethanol through several times of centrifugation at a speed of 3000 rpm.

2.2.3 Synthesis Silver Nanowires by mixing PVA and PVP as Capping Agent (sample 3-5)

Firstly, mix 0.33 gr of PVA and 0.25 gr of PVP in 10 ml of ethylene glycol using an Erlenmeyer flask. Then, 40 mL of µl of a 0.1 M NaCl/EG solution was added into the solution and stirred for one hour. After that, eight mL of a 0.3 M $AgNO_3$/EG was added dropwise for 5-10 minutes followed by 350 rpm and at a constant temperature of 130°C and 90 minutes. Furthermore, the solution was taken out and let to cool at room temperature. The solution centrifuged at 3000 rpm and 30 min each with ethanol. The final product preserved in ethanol until characterization.

2.3 Characterization

UV-vis spectrometer (Shimadzu, UV-1700) was used to measure the absorption spectrum of silver nanowires solution in the wavelength range of 300 to 700 nm.The morphology and size of silver nanowires were observed using scanning electron microscopy (JEOL, JSM-6510) by accelerating voltage of 10 kV. Furthermore, the crystal structure of silver nanowires was analyzed using XRD

(Shimadzu R6000) by CuKα (λ = 1.54184 Å) with a scanning 2θ in the range of 20° to 90°.

3. Results and Discussion

3.1 The Formation of Silver Nanowires by using PVA and PVP

The formation of silver nanowires explained as follows. In the polyol process, the formation anisotropic of Ag nanostructures occurs through two major steps[30]. The first step, Ag^+ ions from a precursor solution reduced to the Ag atoms, which then combine to form nuclei. Thermal energy due to crystal defects and subsequently forming nanostructures with specific morphology when the nuclei growth. It will be decomposition of Ag particles formed in the reaction with the oxygen (O_2) contained in the air. The reduction of Ag^+ ions will compete with the decomposition of oxidation on Ag, so that the nucleation and growth of NPs will be slow during the reaction[31]. It starts due to the heating process of ethylene glycol at high temperature and reducing the silver ion into neutral silver as shown by Formula (1)[32–34].

Formula (1):

$$2HOCH_2\text{-}CH_2 \xrightarrow{130\,^oC} 2CH_3CHO + 2H_2O$$

The glycolaldehyde (CH_2CHO) resulted from the above process was then used to reduce $AgNO_3$ to Ag^+ ions and Ag atoms as shown in Formula (2) and Formula (3).

Formula (2):

$$2CH_3CHO + 2AgNO_3 \rightarrow CH_3CO\text{-}OCCH_3 + 2Ag^+ + 2HNO_3$$

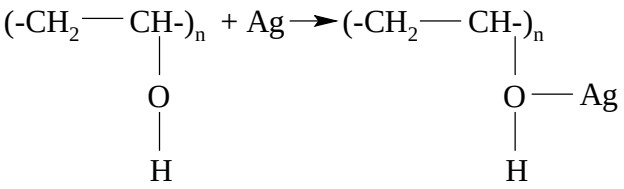
Formula (3):

$$2CH_3CHO + 2Ag^+ \rightarrow CH_3CO\text{-}OCCH_3 + 2Ag + 2H^+$$

The synthesis methods in solution phase and the forming process of the metal nanocrystal can be explained by the growth of metal from a neutral state. In this approach, there are several stages that occur during the synthesis process, the process of nucleation, growth of nuclei become the seeds, and becomes growth of nanocrystal. In the solution phase, metal nanocrystal resulted with different shapes and sizes due to several factors. The first factor, the equilibrium condition will not be achieved during the synthesis process. Furthermore, the surface energy of different side of nanocrystal will be different due to the anisotropic interactions with capping agent and solvent. Moreover, the twin defect will be formed during the nucleation process and will produce a specific shape due to high temperatures during the synthesis process[35].

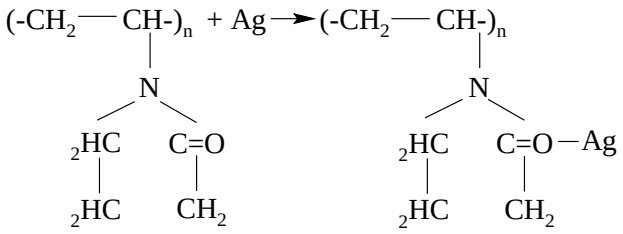
Ethylene glycol is used for the reduction of Ag cations. Ethylene glycol serves as a solvent for PVA, NaCl and $AgNO_3$ powder. Ethylene glycol has a boiling point of 197°C. During the reaction process, the color of the solution turns from pale white to light brown, red, dark gray and eventually gray-green[36] . In the initial step, the ethylene glycol is heated at high temperatures to be converted to glycolaldehyde function to reduce Ag ions[10]. When the chloride ions added, Ag atoms are formed through a process of electrostatic stabilization then formed Ag seeds. The presence of PVA and PVP will cause Ag seeds adsorbed onto the surfaces of Ag through the Ag-O bonding. The role formation of silver nanowires using PVA as a capping agent is described by Formula (4)[12,33,37].

Formula (4):



During the capping process, a low concentration of silver nanoparticles was also capped through a bonding of Ag-O. In this condition, the structure grew up to become MTPs seeds. Due to the capping and the bonding of MTPs of Ag by PVA, then the particles grew up into nanowires. The hydrogen ion (H^+) from hydroxyl group (OH) of PVA disturbed the formation of MTPs into silver nanowires. PVP has the advantage affinity to many chemicals to form coordinative compounds. PVP has a polyvinyl skeleton with a strong polar group (ring pyrrolidone). The polar group (C = O) will interact with Ag^+ ions and form a coordination complex compound as in Equation (2). As well as PVA, the Ag-O bonding formed when the PVP as a capping agent interacting with Ag seeds. Similar to PVA, the role of PVP as a capping agent is described by Formula (5).

Formula (5):



One-dimensional (1D) nanostructures in the form of a pentagonal cross section can grow anisotropic decahedral of seeds Ag[24]. Silver nanowires produced through anisotropic growth of MTPs through the interaction between the capping agent or a surfactant and a crystal Ag facets strongly. MTPs growth is the first step to identify the growth of 1D nanostructures of Ag. MTPs (with octahedral shape) have a configuration five-fold symmetry surface is bounded by {111} facets. To fill the space of 5-fold single crystal, it takes a set of 5-fold twinned particulate decahedral. A twin boundary can represent the highest energy on the surface of the MTPs,it helps to attract surrounding the silver atoms to growth nanowires. The side surfaces of silver nanowire composed of {110} facets[13].

3.2 UV-vis Analysis

Figure 1 shows the UV-vis spectrometer typical of silver nanowires in an ethanol solution. UV-vis spectrometer used to observe the absorbance peaks of silver nanowires. The absorbance peaks of UV-vis spectroscopy can be used to observe the evolution of morphology and size in the growth of silver nanowires structure in different wavelengths.

The spectrum peaks of silver nanowires show two peaks which are relatively sharp absorbance around 350 and 390 nm. The quadrupole resonance excitation of silver nanowires marked at 350 nm, while another peak with high intensity around of 380 nm associated with the absorbance of silver nanowires. The peak around 600 nm will disappear when ethylene glycol solution into the reaction system. The absorbance peak at a wavelength of about 410 nm shift to 380 nm. The absorption peak around 380 nm decreases with increasing number of ethylene glycol at the start of the reaction. Based on the literature, the absorption peak of silver nanowires retained from about 350-380 nm[16,38].

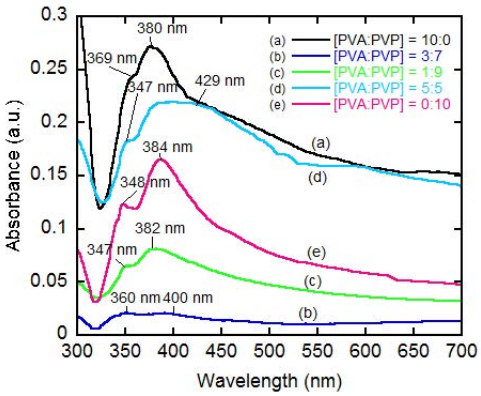


Figure 1. UV-vis spectra of silver nanowires by using PVA and PVP.

3.3 Microstructure Analysis

Figure 2 shows SEM images of silver nanowires for pure PVA as capping agent and stabilizer (sample 1). Figure 2 shows that silver nanowires formed when synthesized using PVA with a diameter of about (190 ± 10) nm and (80 ± 10) μm, respectively. High mechanical strength and low permeability membrane of PVA causes MTPs formed with a longer. PVA caused Ag seeds more capped and formed a large size to form MTPs. The large size of MTPs causes large diameter on the formation of silver nanowires. PVA as surfactant not only capped the {100} facets but also the provision of PVA too much can cause capped the {111} facets. This conditioan can be prevented the isotropic growth of silver nanowires to forming nanoparticles. PVA can't be complete to cover the {100} facets because it blocked with the {111} facets during the growth of the nanowires. This condition is not allowed (forbidden condition) because MTPs can't grow into silver nanowires through isotropic growth.

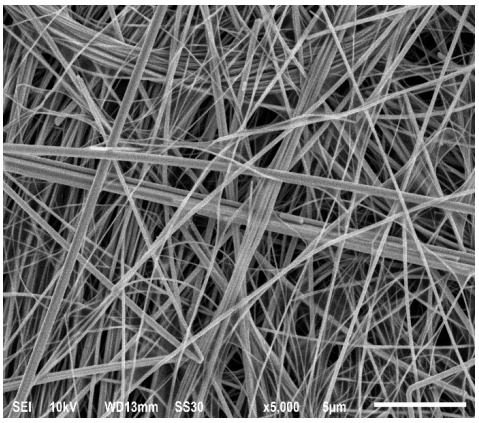


Figure 2. SEM images of silver nanowires by using PVA.

Figure 3 shows the average diameter of silver nanowires about (90 ± 10) nm with a length of (7 ± 2) μm (sample 2). PVA and PVP as a capping agent will be easier to limit the (100) facets compared to the (111) facets of nanoparticles. Ag atoms tend to direct the growth of the (111) facets, it causes elongated decahedron and forms a pentagonal crystal structure. These conditions can lead to a process of continuous growth of nanowires on the (111) facets[24]. The diameter and length of the nanowires can control by changing the molar ratio of PVA and PVP and reaction time by our results as shown in Figure 1 (f). From the SEM images the average nanowires diameter decreases with the presence of PVP.

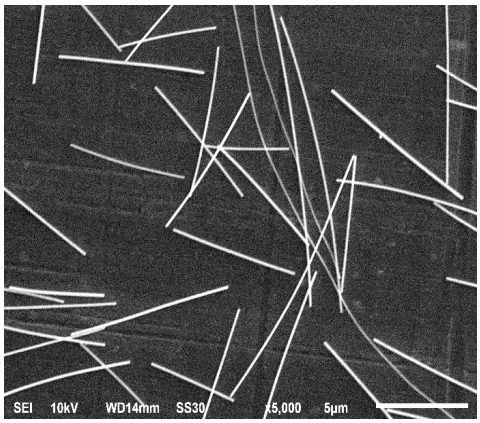


Figure 3. SEM images of silver nanowires by using PVP.

For the molar ratio of PVA and PVP are balanced (sample 3), a lot of MTPs formed with micrometer size Figure 4. The diameter and length of silver nanowires decreased about (400 ± 130) nm and (2 ± 1) μm, respectively. For the molar ratio of [PVA:PVP] is 3:7 (sample 4) as shown in Figure 5. The diameter of silver nanowires is about (300 ± 100) nm and (8 ± 2) μm in length. The diameter size of silver nanowires is continued to decrease with decreasing volume of PVA. Decreasing in the volume of PVA can reduce the mechanical strength of the capping agent on the synthesis silver nanowires. These conditions affect reduction the number and size of the MTPs and nanowires.

For the molar ratio of [PVA:PVP] = 1/9 (sample 5), the diameter and length of silver nanowires about (180 ± 35) nm and (10 ± 5) μm, respectively as shown in Figure 6. The same condition occurs when not given a mixture of PVA. To get the Ag nanowires with pentagonal cross section and high yield, the molar ratio of PVA and PVP should be controlled. The growth rate of Ag atoms must be controlled by the addition of a chloride ion to reduce oxidative etching of MTPs during synthesis. Chloride ions play a critical role as a coordinating ligand in the synthesis silver nanowires. Chloride ions will react with Ag^+ ions to produce a colloidal of AgCl by decreasing the concentration of Ag ions in the solution.

PVA as a capping agent in the synthesis AgNWs can enlarge the diameter AgNWs because PVA has a high mechanical strength. The etching process of MTPs Ag seeds difficult to occur because the presence of PVA has coated it. This condition causes the MTPs Ag seeds has a large size. The mechanical strength of the capping agent is adecline when to reduce the volume of PVA. Declining of mechanical strength of the capping agent causing etching process Ag nanostructures more controllable to growth of silver nanowires with a smaller diameter.

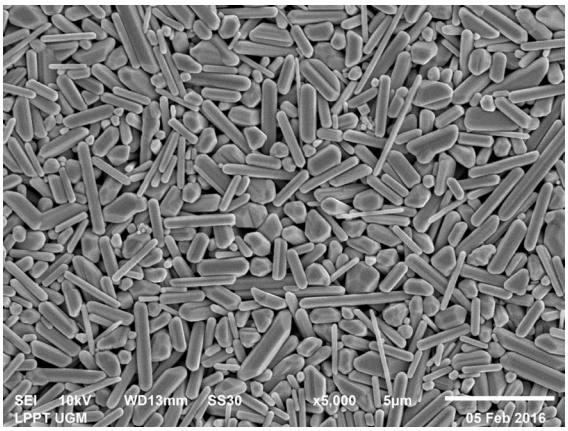


Figure 4. SEM images of silver nanowires by using PVA and PVP for molar ratio [PVA:PVP] of 5:5.

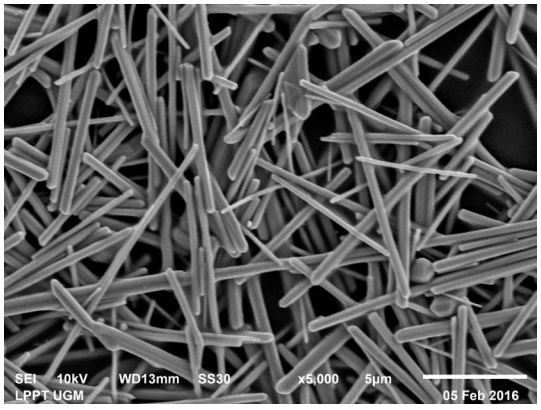


Figure 5. SEM images of silver nanowires by using PVA and PVP for molar ratio [PVA:PVP] of 3:7.

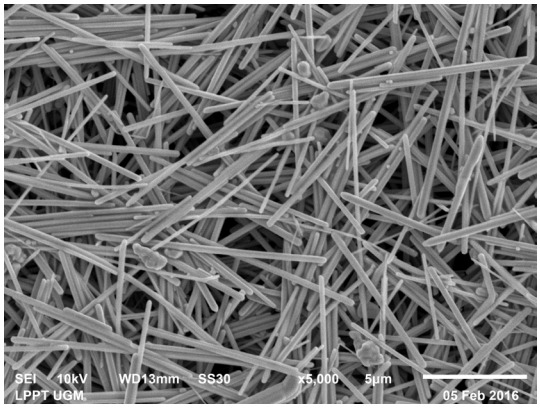


Figure 6. SEM images of silver nanowires byusing PVA and PVP for molar ratio [PVA:PVP] of 1:9.

3.4 Crystal Structure Analysis

The XRD pattern of silver nanowiresdepicted in Figure 7. The crystal structure of silver nanowires depiced five diffraction peaks from XRD pattern analysis by using PVA and PVP as a capping agent. All peaks can index to cubic phase Ag, which each angle of diffraction 2θ was at 37.58° (111), 43.77° (200), 63.98° (220), 76.96° (311) and 81.11° (222) for a capping agent of PVA. For PVP as capping agent, the peaks diffraction 2θ were at 38.25° (111), 44.43° (200), 64.55° (220), 77.51° (311) and 81.65° (222).

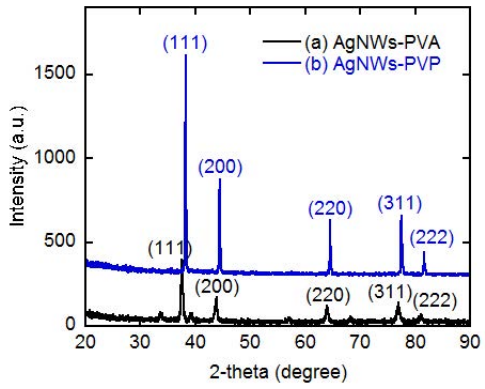


Figure 7. XRD pattern of silver nanowires of PVA and PVP.

The number in the parenthesis indicates the corresponding crystal plane. According to standard JCPDS card of 04-0783 from ASTM, the XRD pattern indicates that the silver nanowires were crystallized. The crystalline of the silver nanowires can identify as a face-centered cubic (fcc). The silver nanowires synthesized in this study also showed a high aspect ratio. This case showed the diffraction signal peak of XRD at (111) was larger than those at (200), i.e. about 2-fold. The calculated lattice constant according to the spacing distance (d_{hkl}) of the (111) planes was 4.1454 Å for PVA and 4.0756 Å for PVP as a capping agent. This calculated lattice constant is very close to the literature value of 4.086 Å[19,21,39].

4. Conclusion

One-dimensional Ag nanostructures with uniform diameters and high aspect ratios grown have been synthesized by polyol process using PVA and PVP as a capping agent. The spectrum peaks of silver nanowires show two SPR peaks around of 350 and 390 nm. From the XRD analysis, the crystalline of the silver nanowires can be identified as a face-centered cubic. The diameter and length of silver nanowires could be adjusted from approximately 100 to 500 nm and 10 to 90 µm, respectively. The molar ratio of PVA and PVP play an important role for controlling the shapes and sizes of silver nanowires. The silver nanowires synthesized by PVA stronger and not easily broken.

5. Acknowledgment

This work is supported by a research grant of "International Research Collaboration and Scientific Publication, Contract No. LPPM-UGM/998/LIT/2015" by the Directorate General of Higher Education (DIKTI), Ministry of Education and Culture, the Republic of Indonesia.

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Indian Journal of Science and Technology

Country	India
Subject Area and Category	Multidisciplinary Multidisciplinary
Publisher	Indian Society for Education and Environment
Publication type	Journals
ISSN	09745645, 09746846
Coverage	2012-2016 (cancelled)

25

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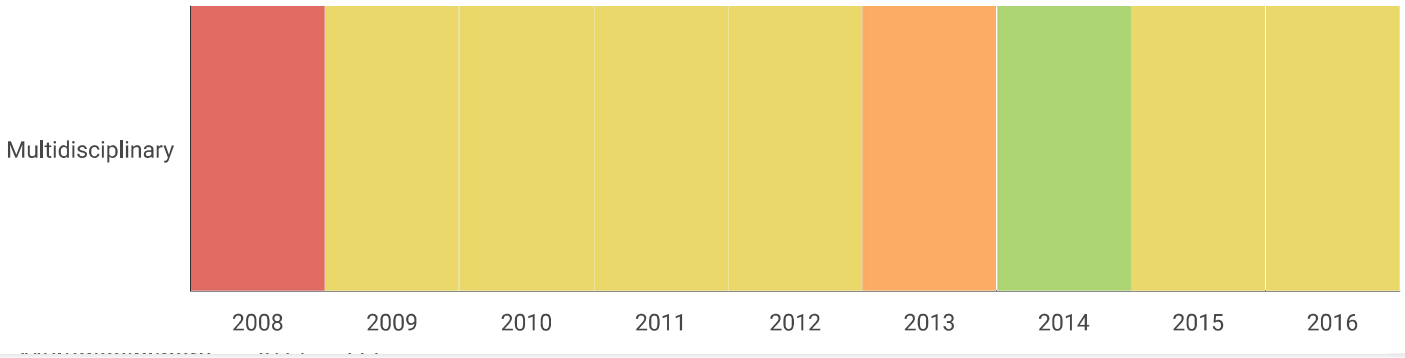

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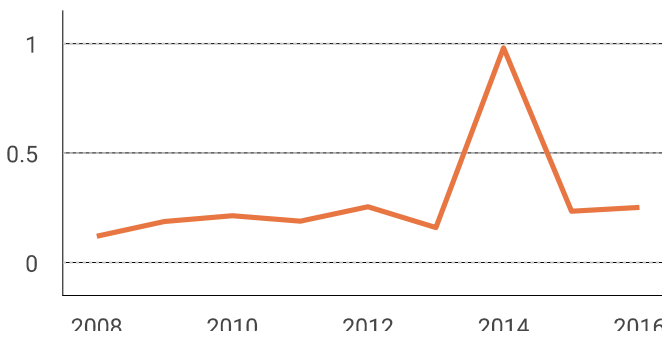

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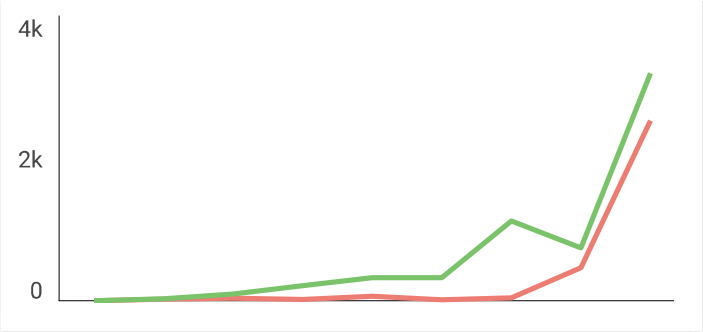

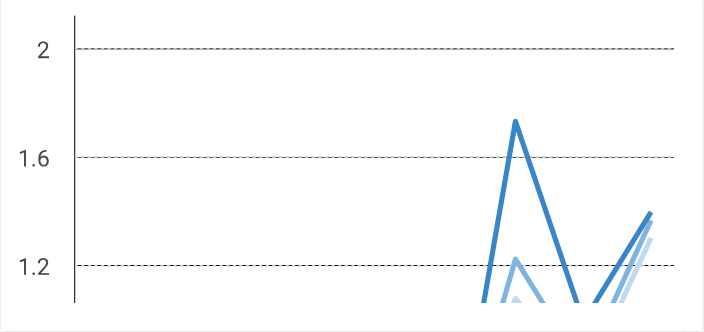

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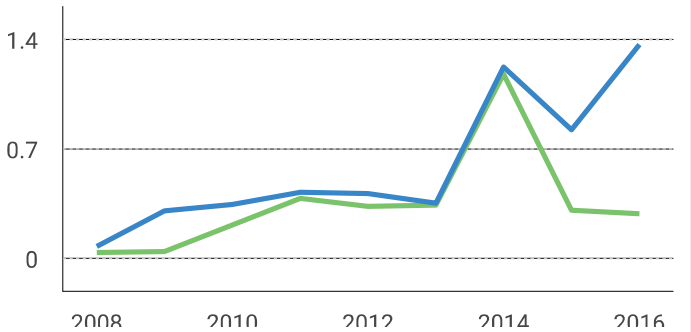

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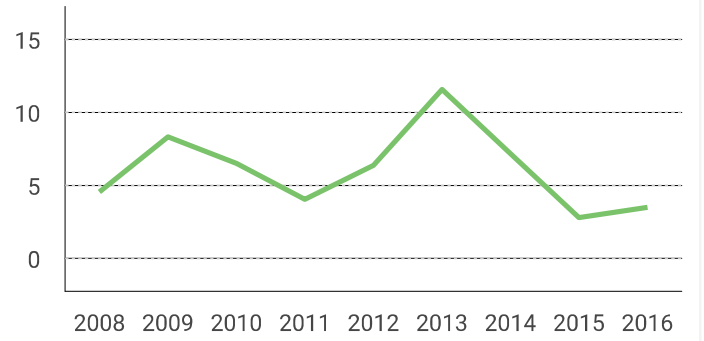

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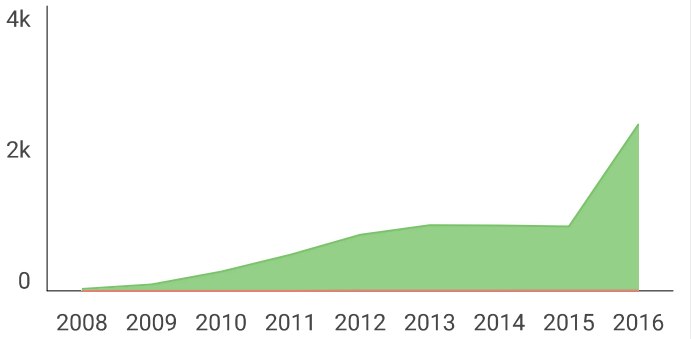

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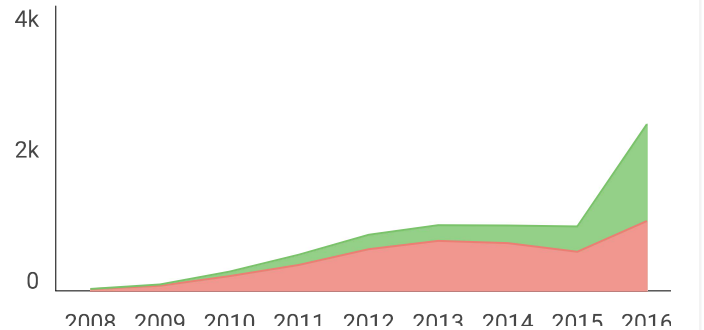

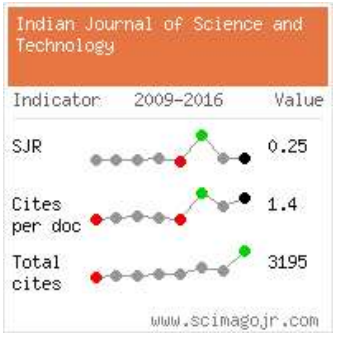

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